

3-24-93  
E-7456

NASA Technical Memorandum 105939

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March 1993

**NASA**

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## SUMMARY

Sliding durability studies of candidate ceramic fibers were conducted in hydrogen to support the high temperature seal development program at NASA Lewis Research Center. Pin-on-disk tests were used to measure the friction and durability of a tow or bundle of ceramic fibers in sliding against a superalloy disk. This procedure was used previously to test candidate fibers in an air environment.

The fibers based upon mullite ( $\text{Al}_2\text{O}_3\text{-SiO}_2$ ) chemistry (Nextel 550, 440, and 312) exhibited better durability in hydrogen than in air. HPZ, a complex silicon carboxynitride fiber which showed good durability in air, however, showed a significant loss of durability in hot hydrogen. These results are consistent with recent thermodynamic and experimental studies of ceramic compatibility with hydrogen at elevated temperatures.

These research results indicate that only oxide fibers display good durability in both air and hydrogen environments. Also, simple, low cost testing in air can provide an adequate data base for initial seal material screening and selection, especially for oxide fiber candidates. The findings of this research provide critical input to the seal design team.

## INTRODUCTION

Numerous technical challenges must be overcome to successfully develop hypersonic flight vehicles such as the National AeroSpace Plane, NASP. Many of these are related to the harsh environmental conditions encountered by airframe and engine seals. Frictional heating by the hypersonic airstream and combustion heating from the  $\text{H}_2$  fuel/air mixture necessitate the design and utilization of seals and materials capable of operating near or above 1000 °C (ref. 1).

Novel seal designs have been developed at NASA Lewis Research Center to address high temperature dynamic sealing applications on hypersonic vehicles. One design approach uses a flexible woven rope seal to prevent leakage between adjacent panels which must articulate relative to one another, for example, engine sidewall seals (fig. 1). To minimize seal actuation forces and seal damage, the tribology (friction and wear behavior) of the system is a critical design issue.

To understand the tribology of potential seal designs, research has been conducted to assess the sliding friction and durability of candidate ceramic rope seal fiber materials. Previous testing has been conducted on various silicon oxide and carboxynitride (C, O, N containing) ceramic fibers in air using a modified pin-on-disk apparatus (refs. 2 to 4).

Because some sealing applications may encounter hydrogen as well as air (engine seals for example), the performance of candidate seal fibers in hydrogen is also of importance. The following paper describes pin-on-disk testing of seal fibers in hydrogen from 25 to 900 °C to help identify leading candidate materials for further study and engineering consideration.



## MATERIALS

The materials tested in this test program consist of four candidate fiber materials and one disk counterface material. The fibers tested were 3M's Nextel 312, Nextel 440, and Nextel 550, and Dow Corning's HPZ. The fiber composition and mechanical properties are given in detail in table I. The Nextel 312 and 440 fibers are made from alumina, boria, and silica. Nextel 312 contains 15 wt % of the glass former, boria, Nextel 440 contains only 2 wt % boria. Nextel 312, therefore, is more amorphous and glasslike and also has, in general, lower tensile strength than Nextel 440. Nextel 550 is similar to 440 but only contains alumina (73 wt %) and silica (27 wt %). HPZ is a complex silicon carboxynitride fiber containing 57 wt % Si, 28 wt % N, 10 wt % C, and 5 wt % O. The Nextel fibers tested were slightly oval in cross section with diameters of about 11  $\mu\text{m}$ . The HPZ fibers had a round cross-section with a diameter of about 12.0  $\mu\text{m}$ . All of the samples were heat cleaned at 500 °C, in air for 1 hr to remove an organic sizing compound used by the manufacturer during processing (ref. 5).

In addition to the heat cleaning step, some Nextel samples, which are designated with an HT suffix, Nextel 440HT for example, were heat treated in air at 950 °C for 12 hr prior to testing. This heat treatment is reported to make some of the fibers more resistant to moisture degradation at temperature (ref. 6) and serves as a method to assess the high temperature resistance of the fibers. In previous sliding durability testing in air, the heat treatment reduced the sliding durability of Nextel 312 and 440 at elevated temperatures but had no significant effect on Nextel 550.

The candidate fibers are selected for  $\text{H}_2$  testing because they were tested previously in air (refs. 2 to 4) and exhibited good durability behavior except Nextel 312 which lacked high temperature wear resistance. Nextel 312 is included here as a representative baseline fiber rather than a potential fiber candidate.

The test disks were made of Inconel 718, a precipitation hardened nickel-chromium alloy. Table II lists the composition and hardness of the disk material. This material is being considered as a possible candidate for seal applications because of its high temperature strength and oxidation resistance. Prior to testing, the disk surface is lapped with alumina abrasive to a surface finish of about 0.1  $\mu\text{m}$  rms. After lapping, the specimens are cleaned with freon, ethyl alcohol, scrubbed with paste of levigated alumina and water, rinsed with deionized water, and air dried.

## PROCEDURES/APPARATUS

The candidate fibers are tested in sliding using a pin-on-disk apparatus. This testing technique, which has been described elsewhere (ref. 2) consists of wrapping a bundle of fibers (typically 6000,  $\approx 10\text{-}\mu\text{m}$  diameter fibers) over the tip of a specially machined Inconel 718 pin. The pin has grooves machined into its tip and shank to accept the bundle and prevent its slipping off during testing (fig. 2). In addition, a 3.2-mm diameter flat spot is machined on the pin tip to provide a uniform sliding area. The bundle is given a one half or 180° twist across the flat contact spot to help contain the bundle in the sliding contact and to orient the fibers at approximately a 45° angle with the sliding direction to better simulate proposed braided seal configurations (fig. 3). Figure 4 shows a photomicrograph of a wrapped pin sample prior to testing.

To test a fiber candidate, the pin is slid against a counterface disk in a high temperature pin-on-disk tribometer. The disk is 63.5 mm in diameter and 12.7 mm thick. The pin generates a 51-mm wear track on the face of the disk. Figure 5 shows a schematic of the tribometer used in this work.

The pin-on-disk tribometer heats the specimens using an induction heating coil to achieve the desired test temperatures. For the tests conducted here, the sliding speed was 0.025 m/s and the load was 0.270 kg. Test temperature was 25, 500, and 900 °C and measured using infrared pyrometry and contact thermocouples. The chamber atmosphere during these tests was technical grade hydrogen (nominal composition: 99.95 percent H<sub>2</sub>, N<sub>2</sub> < 400 ppm, H<sub>2</sub>O < 50 ppm, O<sub>2</sub> < 50 ppm) flowing at  $7.1 \times 10^{-3}$  m<sup>3</sup>/min. Prior to introducing H<sub>2</sub> into the chamber, Argon was used for purging. The test chamber pressure was maintained at 2 psig throughout the tests. The test duration was 120 min. Friction was continuously measured during the tests. Wear was quantified after each test using visual and SEM analysis. Previous tests indicated that fiber wear occurs in a fairly uniform manner during the tests. That is, the wear rate is more or less constant over the entire test suggesting that interfiber friction and load sharing effects are small (ref. 2).

## RESULTS

### Friction

The friction and durability results for the various fibers tested are summarized in table III. The friction coefficients for all of the fibers ranged from 0.57 to 0.67 at room temperature, from 0.60 to 0.73 at 500 °C, and from 0.40 to 0.99 during sliding at 900 °C. Nextel 312 and 550 exhibited friction coefficients of  $\approx 0.5$  to 0.7 over the entire test temperature range. Nextel 440 displayed friction similar to Nextel 550 and 312 at 25 and 500 °C but much higher friction coefficients, about 0.9 to 1.0, at 900 °C. HPZ, the only nonoxide fiber tested, exhibited lower friction at 900 °C, about 0.4, than at 25 and 500 °C at which temperatures the friction was about 0.7.

### Durability

The wear behavior of the fibers is quantified using cycles to failure (CTF). The CTF is really a measure of a fiber's wear resistance or durability and is inversely related to the wear rate. The CTF is defined as the number of sliding passes (disk rotations) needed to wear completely through a test bundle of 6000 fibers. The CTF measurement is somewhat arbitrary. That is, it is related to the tests conducted here. Differing test geometries and conditions such as number of fibers in a bundle, loads, etc. may affect the CTF value. However, it is a useful measure for ranking and comparing relative fiber performance.

With the exception of 550HT, fiber durability decreased with increasing temperature. Nextel 550HT showed better wear resistance at 900 °C than at 500 or 25 °C although there is considerable data overlap in the CTF measurements. The uncertainties given in table III represent data scatter from at least three repeat tests for each average data value.

HPZ (Si, C, O, N), the only nonoxide fiber tested in this program, displayed friction and wear properties which significantly differed from the other fibers tested. For example, in contrast to the other fibers, HPZ showed much lower friction at 900 °C than at 25 °C. However its durability at 900 °C was dramatically reduced requiring very early test termination ( $\approx 100$  cycles).

### Wear Surface Morphology

The wear surfaces of the fiber specimen bundles (tows) and counterface disks were examined with a Scanning Electron Microscope, SEM, to better understand the measured wear behavior. Figures 6 to 9 show both oxide fibers and HPZ after sliding at temperatures from 25, 500, and 900 °C. At 25 °C, all of



the examined fiber wear surfaces exhibited characteristics of abrasive wear such as small particulate debris and faceted fractured failure surfaces. The disk wear tracks appear smoothed or smeared slightly with no evidence of severe material removal (fig. 10).

At 900 °C, the oxide fiber materials have wear surfaces very similar to those observed at 25 °C except the wear surfaces are smoother suggesting more mild abrasive wear. The complex carbide fiber, HPZ, however, shows evidence of a reaction occurring at the sliding contact. This can be seen as severe blistering and bubbles present on the worn fiber surfaces. Blistering usually indicates the presence of a gas within the fiber being created or evolved and may be a contributing factor to the low durability of HPZ at 900 °C.

### Discussion of Results

Prior to the current durability testing in hydrogen discussed here, these ceramic fibers were tested in air (refs. 2 to 4). The results in air suggested that mechanical properties, such as tensile strength, significantly influence durability. For the oxide fibers, mechanical properties seem to dominate durability performance over the entire test temperature range. But for nonoxide fibers, such as HPZ, mechanical properties have a predominate effect on durability only at room temperature. At elevated temperatures, the fiber durability of HPZ seemed to be affected by tribochemical effects, such as oxide layer growth, as well as mechanical effects (ref. 3).

In hydrogen, similar results are obtained except that durability and friction coefficients for the oxide fibers are generally higher than in air. This difference in durability between H<sub>2</sub> and air can be attributed to the difference in the apparent wear mechanisms which occur. In air, which contains water vapor, the oxide fibers fail by brittle fracture (ref. 2). This is consistent with research by others on ceramics which suggest that water molecules degrade the atomic bonding in ceramics and accelerate cracking (ref. 7). Therefore, when tested in moist air, the fiber wear behavior is dominated by fracture.

In contrast, when the oxide fibers are tested in dry H<sub>2</sub>, the dominant wear mechanisms appears to be abrasion. Abrasive wear, in general tends to be a gradual wear process as opposed to fracture which is catastrophic and can be very rapid. The different wear mechanism(s) observed in the different environments may account for the higher durability of the oxide fibers in H<sub>2</sub>.

HPZ, the nonoxide fiber, exhibited very low durability in H<sub>2</sub>. Recent experimental and theoretical thermodynamic stability research suggests that environmental interactions with the fibers may have an influence on durability (ref. 8). Reference 8 describes a thermodynamic analysis to predict the chemical stability of a wide variety of ceramic materials including oxides, carbides and nitrides in H<sub>2</sub>, from 25 to 1300 °C. Experimental studies were carried out in reference 9, to validate the thermodynamic predictions. These analyses of ceramic stability can be applied to the durability behavior of the ceramic fibers tested in the present work.

For example, in reference 8, Misra et al. conclude from a purely thermodynamic standpoint, that alumina based oxide ceramics offer the most potential for stability in hot hydrogen environments. Furthermore, their analyses suggest that carbides and nitrides of silicon, which make up the complex structure of HPZ, are likely to suffer reactions with a hot H<sub>2</sub> environment.

Follow-on experimental work supports the findings of the thermodynamic studies (ref. 9). Oxide ceramics based upon alumina and mullite (such as Nextel 550) offer good intrinsic stability with hydrogen; whereas, silicon carbide and nitride rely upon SiO<sub>2</sub> passivating layer for protection. In the sliding tests conducted here, it is plausible that the passivating SiO<sub>2</sub> layer is being disrupted by the wear process

leading to enhanced attack of the HPZ by the hydrogen. The wear features, observed for HPZ tested in  $H_2$  at 900 °C, namely blistering shown in figure 9, suggest a reaction occurs creating a gas. A possible reaction presented by Misra is the production of methane,  $CH_4$ , from the carbon in the fibers and the  $H_2$  test gas. Although the exact reaction/degradation mechanism(s) is not known at this time, the changes observed at the sliding interface (i.e., blistering) may explain the significant drop in high temperature durability of HPZ fibers.

### Effect of Heat Treatment

Previous durability testing of boria ( $B_2O_3$ ) containing ceramic fibers (Nextel 312 and 440) ceramic fibers in air indicated that static thermal exposure to air at 950 °C for 12 hr significantly reduced their subsequent sliding durability (ref. 2). Nextel 550 fibers which contain only  $Al_2O_3$  and  $SiO_2$  did not suffer this durability reduction after the thermal exposure. In fact, Nextel 550 exhibited slightly improved durability at 900 °C following the static thermal exposure (ref. 4) at 950 °C for 12 hr. The reasons for these durability changes are unclear but may be related to the interaction of the  $B_2O_3$  and the environment and the crystal structure of the fibers (ref. 10).

When tribotested in hydrogen, the  $Al_2O_3$ - $SiO_2$ - $B_2O_3$  fibers (Nextel 312 and 440) showed no such degradation in durability following the same thermal exposure. Nextel 550, the  $Al_2O_3$ - $SiO_2$  fiber, behaved in  $H_2$  much in the manner that it behaved in air, showing slightly improved high temperature durability following the thermal exposure although there was considerable data overlap. Therefore, it is apparent that there was some interaction between an air test environment and the fibers containing  $B_2O_3$ , which affected their durability at elevated temperatures in air. The exact effect is not understood at this time. However, the data suggest that, when tested in  $H_2$  the thermal exposure creates no significant durability degradation.

### CONCLUSIONS

1. When tested in  $H_2$ , oxide based fibers such as  $Al_2O_3$ - $SiO_2$ - $B_2O_3$  (Nextel 312 + 440) and  $Al_2O_3$ - $SiO_2$  (Nextel 550) exhibited better durability than when tested in air.
2. When tribotested in  $H_2$  following a static heat treatment in air, the oxide fibers do not suffer a sliding durability degradation as was observed in air tribotesting.
3. The wear mode in  $H_2$  for all of the oxide fibers tested at 25 °C appears to be abrasion leading to fracture due to sliding, whereas HPZ exhibits more severe brittle fracture behavior at 25 °C. At elevated temperatures, the oxide fibers continue to wear by mild abrasion. The carbide fiber, HPZ, however seems to react with the  $H_2$  test gas exhibiting increased wear.
4. When tested in  $H_2$ , both the Nextel 440 and 550 exhibit good durability properties. However, because Nextel 550 has superior durability in air, it is recommended for further study.
5. Although the carbide fiber, HPZ, exhibited adequate, and stable durability properties in air, its low durability in  $H_2$  along with an apparent reactivity problem make it an unsuitable seal candidate for environments containing  $H_2$ .



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TABLE I.—FIBER COMPOSITION AND MECHANICAL PROPERTIES

Fiber material	Composition, wt %	Diameter, $\mu\text{m}$	E, GPa	Filament tensile strength at 25 °C, manufacturer's data, MPa
Nextel 312	$62\text{Al}_2\text{O}_3-24\text{SiO}_2-14\text{B}_2\text{O}_3$	11	150	1700
Nextel 312HT <sup>a</sup>	$62\text{Al}_2\text{O}_3-24\text{SiO}_2-14\text{B}_2\text{O}_3$	11	150	1250
Nextel 440	$70\text{Al}_2\text{O}_3-28\text{SiO}_2-2\text{B}_2\text{O}_3$	11	190	2000
Nextel 440HT <sup>a</sup>	$70\text{Al}_2\text{O}_3-28\text{SiO}_2-2\text{B}_2\text{O}_3$	11	190	1500
Nextel 550	$73\text{Al}_2\text{O}_3-27\text{SiO}_2$	11	190	2200
Nextel 550HT <sup>a</sup>	$73\text{Al}_2\text{O}_3-27\text{SiO}_2$	11	190	2200
HPZ	57Si 28N 10C 5O	12.6	176	1625

<sup>a</sup>HT designates fibers which have been heat treated (exposed) in air at 950 °C for 12 hr.

TABLE II.—NOMINAL COMPOSITION AND HARDNESS OF INCONEL TEST DISK SPECIMENS

Property	Value
Composition, wt %	70 Ni, 16 Cr, 7.5 Fe, 2.5 Ti, 1 Al, 1 Co, 1 Mn, 0.1 C, and 0.9 other
Hardness, Rockwell C	RC34



TABLE III.—FRICTION AND DURABILITY SUMMARY IN HYDROGEN

Fiber material	Composition, wt %	Diameter, $\mu\text{m}$	$E$ , GPa	25 °C		500 °C		900 °C	
				$\mu$	CTF <sup>4</sup>	$\mu$	CTF	$\mu$	CTF
Nextel 312	62Al <sub>2</sub> O <sub>3</sub> -24SiO <sub>2</sub> -14B <sub>2</sub> O <sub>3</sub>	11	150	0.64±0.15	9200±2500	0.68±0.13	4300±500	0.68±0.14	410±140
Nextel 312HT	62Al <sub>2</sub> O <sub>3</sub> -24SiO <sub>2</sub> -14B <sub>2</sub> O <sub>3</sub>	11	150	.57±.11	4680±2900	.66±.07	2750±2100	.57±.09	740±270
Nextel 440	70Al <sub>2</sub> O <sub>3</sub> -28SiO <sub>2</sub> -2B <sub>2</sub> O <sub>3</sub>	11	190	.64±.09	21 750±6 000	.62±.05	7400±1900	.93±.28	6700±2200
Nextel 440HT	70Al <sub>2</sub> O <sub>3</sub> -28SiO <sub>2</sub> -2B <sub>2</sub> O <sub>3</sub>	11	190	.62±.11	11 600±5 800	.60±.06	5200±2000	.99±.21	5200±800
Nextel 550	73Al <sub>2</sub> O <sub>3</sub> -27SiO <sub>2</sub>	11	190	.67±.16	14 000±5 000	.72±.05	9400±3000	.65±.16	7500±4000
Nextel 550HT	73Al <sub>2</sub> O <sub>3</sub> -27SiO <sub>2</sub>	11	190	.67±.12	6962±2500	.61±.03	4800±800	.56±.20	13 400±4 200
HPZ	57Si 28N 10C 5O	12.6	176	.67±.07	3303±2047	.73±.09	1754±400	.40±.06	108±40

Notes:

<sup>1</sup>Tests conducted for  $\approx 6000$  fiber bundle in hydrogen, 1 in./s sliding velocity, 270 g load for up to 2 hr.<sup>2</sup>HT designates fibers which have been heat treated (exposed) in air at 950 °C for 12 hr.<sup>3</sup>Uncertainties represent data scatter for at least three repeat tests for each condition.<sup>4</sup>CTF, cycles to bundle failure, represent the number of disk rotations required to wear complete test bundle or tow.

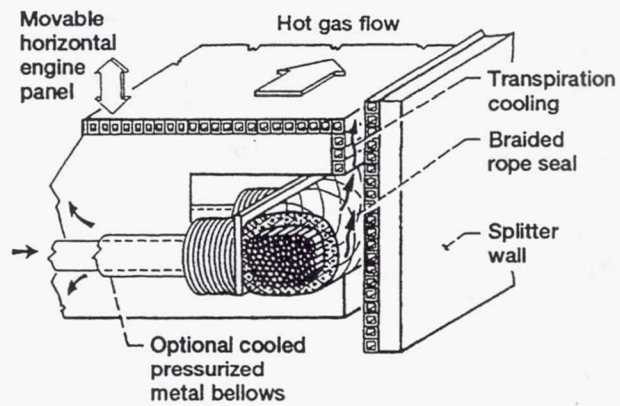


Figure 1.—Cross section of proposed engine seal.

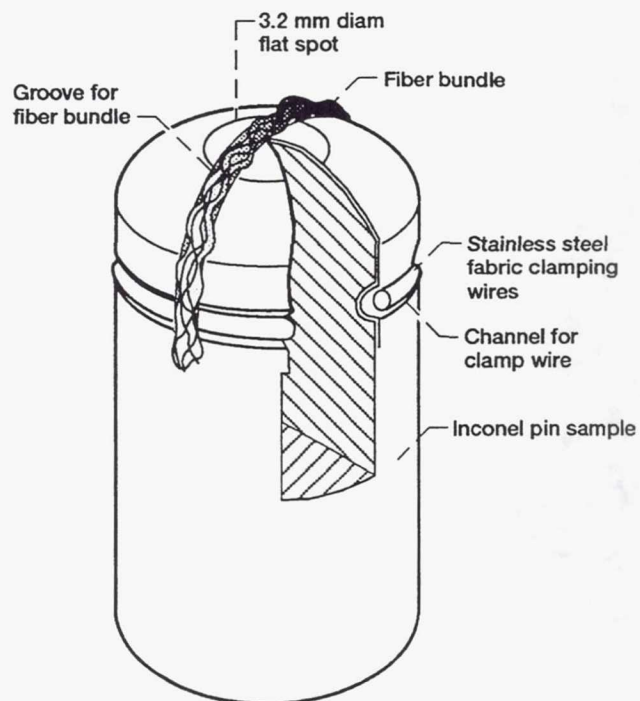


Figure 2.—Pin test specimen.



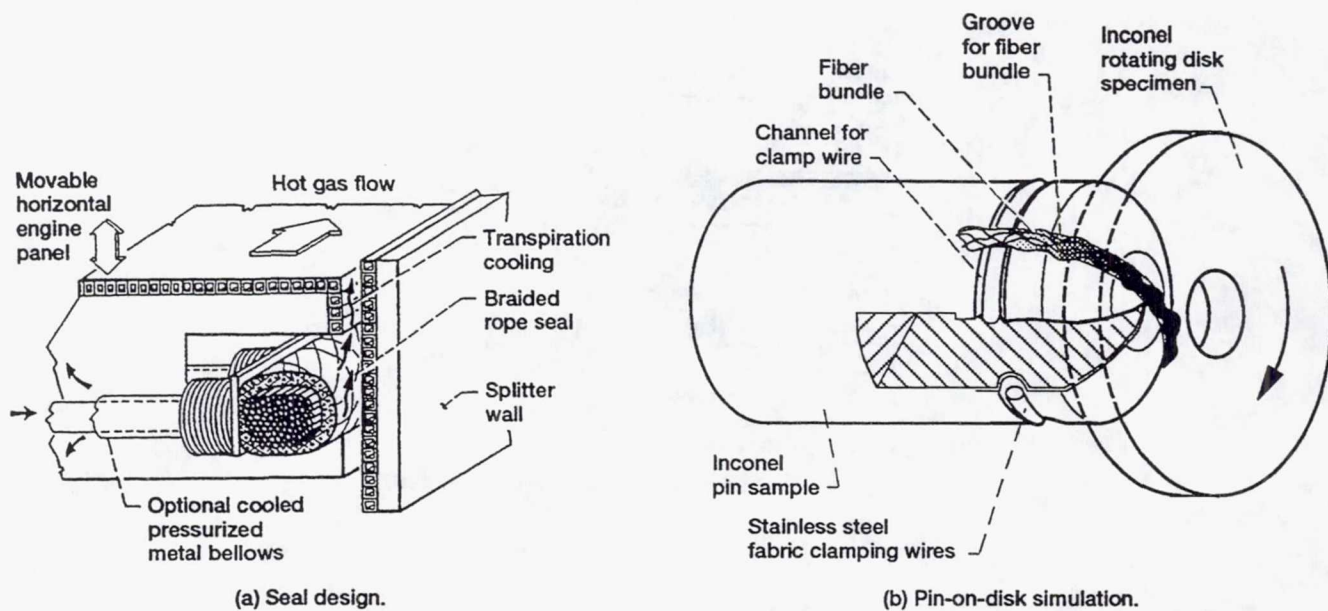


Figure 3.—Fiber tow wrapped over pin is slid against a slowly rotating disk (shown on right) to simulate seal/counterface sliding in application (shown on left).

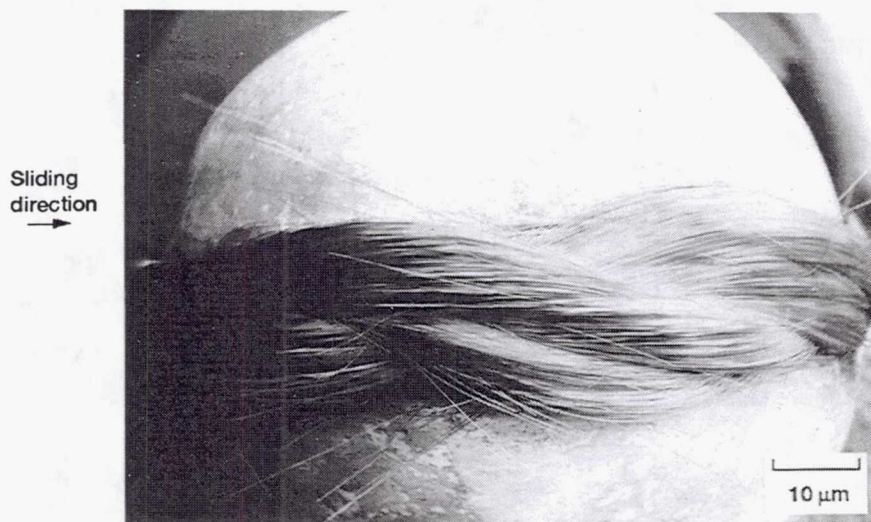


Figure 4.—SEM photomicrograph of fiber-pin specimen prior to testing. Sliding direction is from left to right.

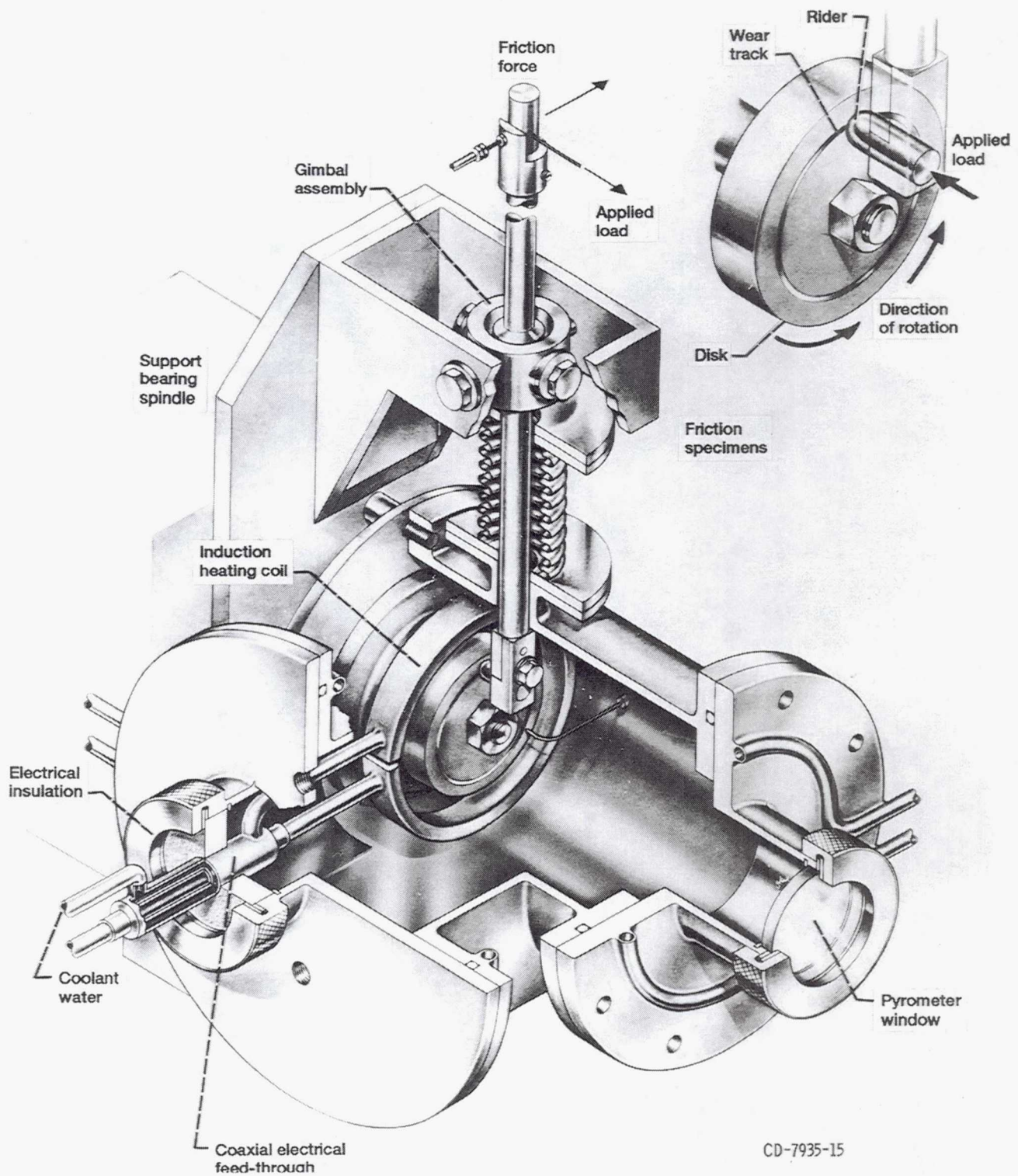


Figure 5.—High temperature pin on disk tribometer.



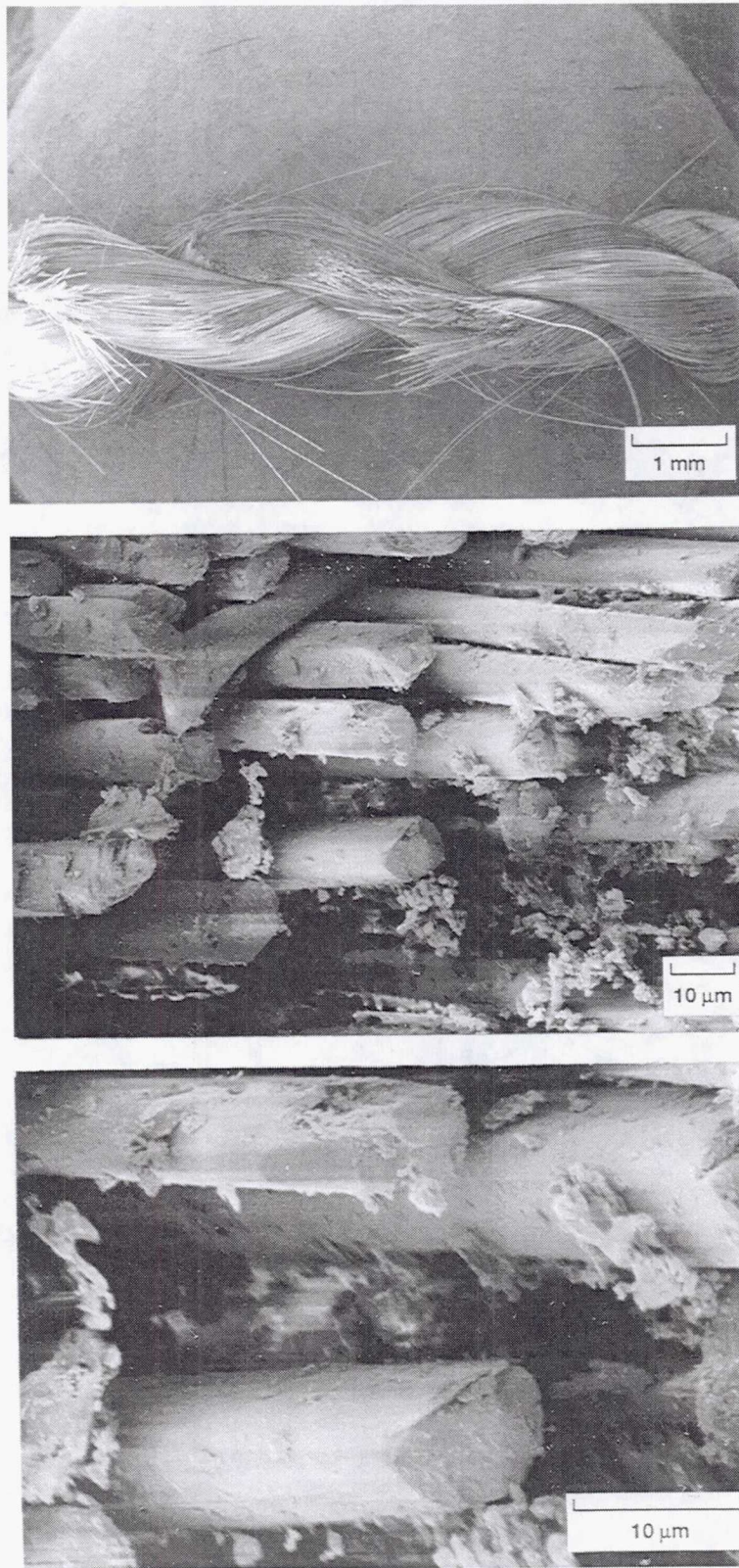


Figure 6.—SEM photomicrographs of  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$ - $\text{B}_2\text{O}_3$ , Nextel 440, fiber after sliding in  $\text{H}_2$  at 25 °C. Surface features include small debris particles suggesting abrasive wear as the degradation mechanism.



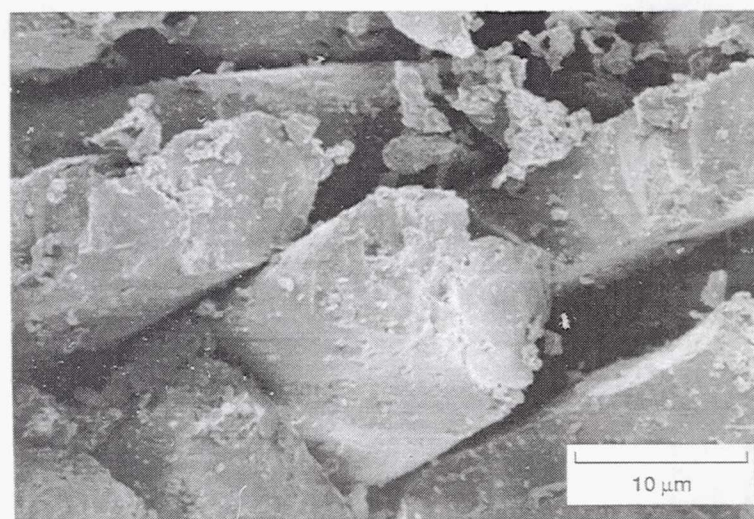
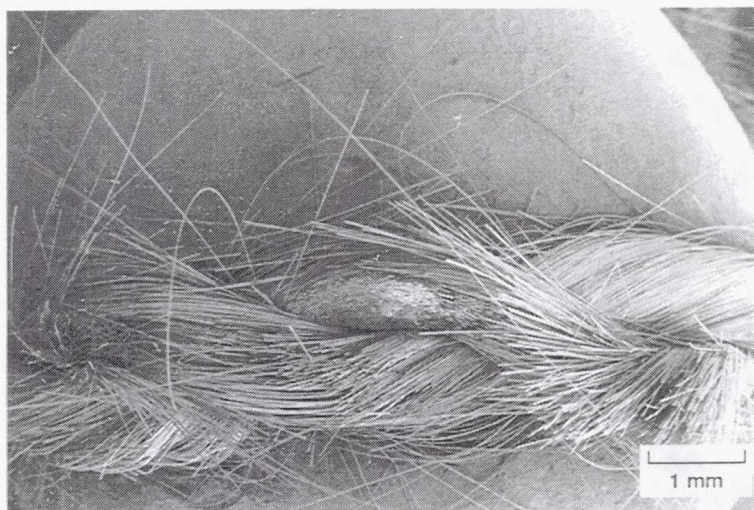


Figure 7.—SEM photomicrographs of complex carboxynitrile fiber, HPZ, after sliding in  $H_2$  at 25 °C. Sliding surface features include abraded regions and faceted fracture regions.



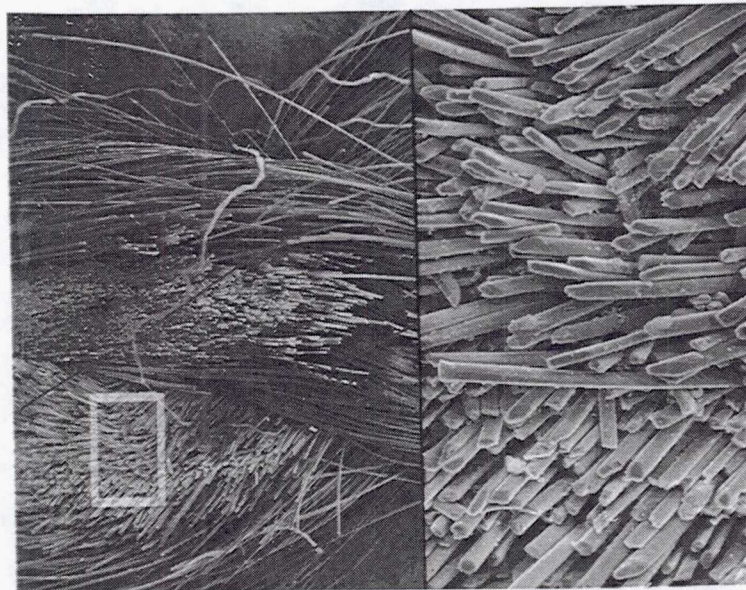
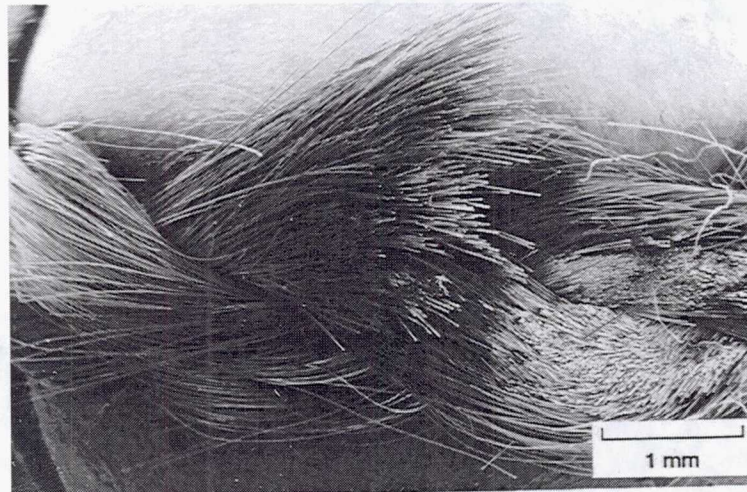


Figure 8.—SEM photomicrographs of  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$ - $\text{B}_2\text{O}_3$ , Nextel 440, fibers after sliding in  $\text{H}_2$  at 900 °C. Smooth, flattened, elliptical wear surfaces and small debris particles suggest mild abrasion occurred.



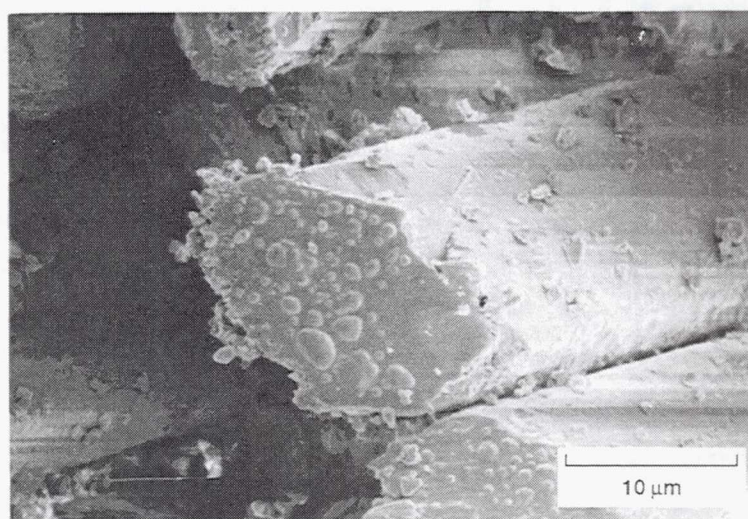
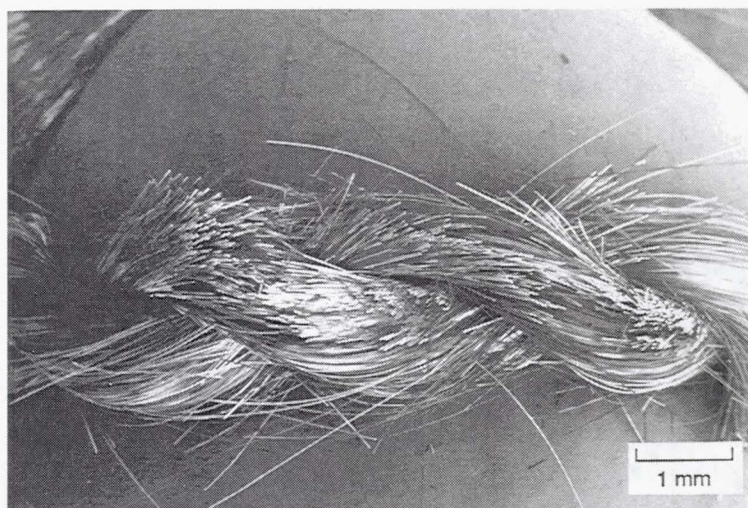


Figure 9.—SEM photomicrographs of complex carboxynitride fiber, HPZ, after sliding in H<sub>2</sub> at 900 °C. Note blistering on sliding surfaces.



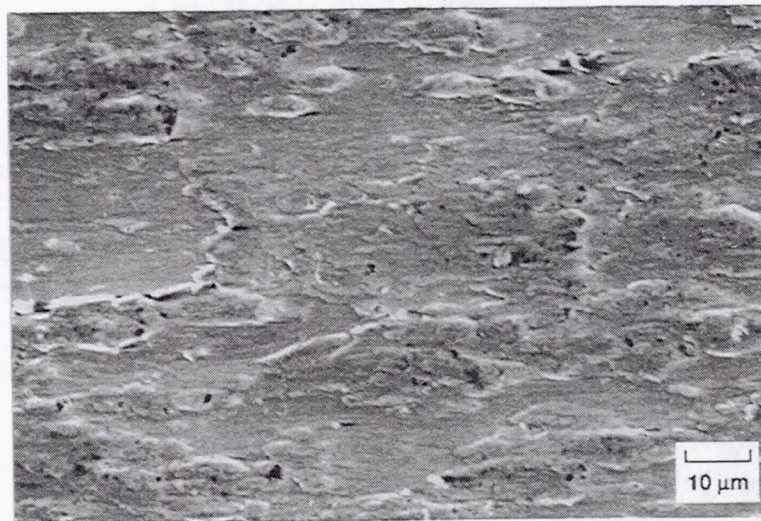
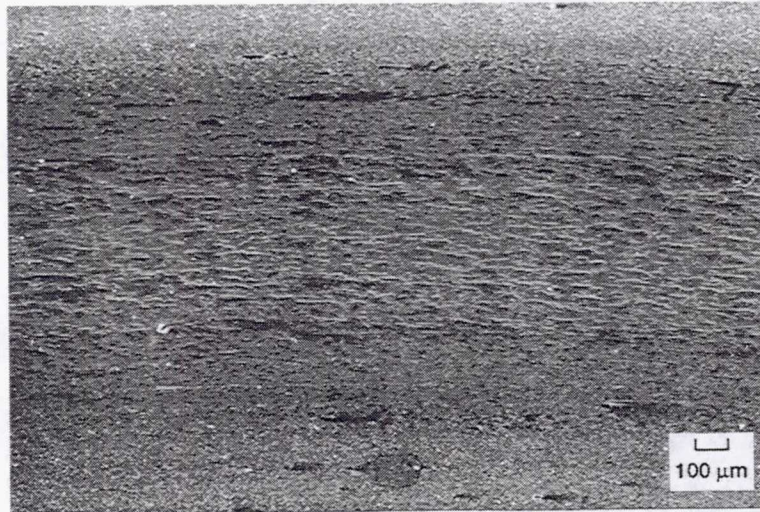


Figure 10.—SEM photomicrographs of Inconel 718 disk wear track after sliding against  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$ - $\text{B}_2\text{O}_3$ , Nextel 440, in  $\text{H}_2$  at 900 °C. Note slightly smeared appearance of wear area.

# REPORT DOCUMENTATION PAGE

Form Approved  
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<b>1. AGENCY USE ONLY (Leave blank)</b>		<b>2. REPORT DATE</b> 1992	<b>3. REPORT TYPE AND DATES COVERED</b> Technical Memorandum	
<b>4. TITLE AND SUBTITLE</b>  Sliding Durability of Candidate Seal Fiber Materials in Hydrogen from 25 to 900 °C			<b>5. FUNDING NUMBERS</b>  WU-505-63-1A	
<b>6. AUTHOR(S)</b>  Christopher DellaCorte and Bruce M. Steinetz				
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b>  National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191			<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b>  E-7456	
<b>9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)</b>  National Aeronautics and Space Administration Washington, D.C. 20546-0001			<b>10. SPONSORING/MONITORING AGENCY REPORT NUMBER</b>  NASA TM-105939	
<b>11. SUPPLEMENTARY NOTES</b> Portions of this report were presented at the 5th NASA/NASP Workshop on Hydrogen-Material Interactions, Scottsdale, Arizona, September 23-25, 1993. Christopher DellaCorte and Bruce M. Steinetz, NASA Lewis Research Center. Responsible person, Christopher DellaCorte, (216) 433-6056.				
<b>12a. DISTRIBUTION/AVAILABILITY STATEMENT</b>  Unclassified - Unlimited Subject Category 23			<b>12b. DISTRIBUTION CODE</b>	
<b>13. ABSTRACT (Maximum 200 words)</b>  Sliding durability studies of candidate ceramic fibers were conducted in hydrogen to support the high temperature seal development program at NASA Lewis Research Center. Pin-on-disk tests were used to measure the friction and durability of a tow or bundle of ceramic fibers in sliding against a superalloy disk. This procedure was used previously to test candidate fibers in an air environment. The fibers based upon mullite (Al <sub>2</sub> O <sub>3</sub> -SiO <sub>2</sub> ) chemistry (Nextel 550, 440 and 312) exhibited better durability in hydrogen than in air. HPZ, a complex silicon carboxynitride fiber which showed good durability in air, however, showed a significant loss of durability in hot hydrogen. These results are consistent with recent thermodynamic and experimental studies of ceramic compatibility with hydrogen at elevated temperatures. These research results indicate that only oxide fibers display good durability in both air and hydrogen environments. Also, simple, low cost testing in air can provide an adequate data base for initial seal material screening and selection, especially for oxide fiber candidates. The findings of this research provide critical input to the seal design team.				
<b>14. SUBJECT TERMS</b>  Seals; Friction; Wear; Hydrogen; Fibers			<b>15. NUMBER OF PAGES</b> 18	
			<b>16. PRICE CODE</b> A03	
<b>17. SECURITY CLASSIFICATION OF REPORT</b> Unclassified	<b>18. SECURITY CLASSIFICATION OF THIS PAGE</b> Unclassified	<b>19. SECURITY CLASSIFICATION OF ABSTRACT</b> Unclassified	<b>20. LIMITATION OF ABSTRACT</b>	



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